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THE STORAGE OF HYDROGEN IN THE FORM OF METAL HYDRIDES -- AN APPLICATION TO THERMAL ENGINES

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16. Abstract
The possibility of using $LaNi_5H_6$, $FeTiH_2$, or MgH_2 as metal hydride storage systems for hydrogen fueled automobile engines is discussed. A study of magnesium copper and magnesium nickel hydrides indicates that they provide more stable storage systems than pure magnesium hydrides. Several test engines employing hydrogen fuel have been developed: a single cylinder motor originally designed for use with air gasoline mixtures, a four cylinder engine modified to run on an air hydrogen mixture, and a gas turbine.

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THE STORAGE OF HYDROGEN IN THE FORM OF METAL HYDRIDES -- AN APPLICATION TO THERMAL ENGINES

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[Translator's Note: This article included a "summary" which was translated into German and Dutch as well as English. All versions conveyed the same content.]

Summary /7*

One of the main problems limiting the use of hydrogen as a fuel is the difficulty of storing it because it is the lightest of all gases (90 g/Nm^3). There are two ways of storing hydrogen industrially: in the form of a compressed gas or as a liquid. The use of metallic hydrides has also been discussed.

Laboratories work primarily with the following compounds: $LaNi_5$, FeTi, and Mg. The table on the next page gives a comparison of the perfomances of compressed gas, liquid, and hydride approaches.

This process does therefore have its interest. From the point of view of volume, $LaNi_5$ incontestably has the best performance with 140 g H_2/l, although with 14 g/kg it is at a disadvantage as regards mass. FeTi, very similar to $LaNi_5$, is no doubt not as costly. /8

As regards on-board systems, the most improtant factor is weight, and here magnesium comes into its own.

We have studied the kinetics of magnesium hydride from the point of view of pressure, temperature, granulometry, and purity of the hydrogen and traced its isothermic pattern. During the

*Numbers in the margin indicate pagination in the foreign text.

		gH_2/kg	Nm^3/kg	gH_2/l	Nm^3/l	2 bar pressure obtained at
$La\ Ni_5\ H_6$	°	14	0.15	140	1.5	20 °C
$Fe\ Ti\ H_2$	°	19	0.2	114	1.25	0 °C
$Mg\ H_2$	°	77	0.85	110	1.2	300 °C
H_2 under 200 bars	°°	14	0.15	18	0.2	
LH_2 at 20,3°K, 1 bar	°°	500	5.5	50	0.5	

° including container

°° excluding container

charge-discharge cycles, we noticed a phenomenon of aging which renders the use of pure Mg difficult. The study of Mg_2Cu and Mg_2Ni showed that this phenomenon can be made to vanish.

We have also given the results of experiments concerning the tranfer of heat and mass obtained with a tank containing hydrides and capable of storing several Nm^3 of hydrogen.

Information gathered from various pieces of literature is also included.

Introduction /9

Ninety grams per cubic meter! This is the incredibly low density of hydrogen, foruteen times lighter than air.

The inflammable air discovered by Cavendish is certainly one of the most studied elements, from its chemical properties to its quantum mechanics.

Today, at the dawn of a new era of energy production opened up by nuclear power, hydrogen is on its way to becoming a universal fuel. Even though world production of hydrogen is already very high and its applications numerous, a fundamental problem remains to be solved: its storage.

Two forms are presently in use: compressed hydrogen and liquid hydrogen.

In its compressed form, this gas is widely used in industry. The progress that can be expected is in the performance per unit mass of the tanks (light alloys, hooped tanks, fiber conglomerates).

In liquid form, the technology has rapidly progressed as a result of space research. Here again, one can expect progress in the areas of structural materials and insulators. In addition, increased efficiency in the liquefaction process will also occur.

Because of the performance per unit mass and volume of these two forms of storage, a third mode is currently experiencing a new surge of interest. This is the storage of hydrogen in the form of metal hydrides.

This technique was already used at the beginning of the century by aeronauts to fill balloons with hydrogen by hydrolyzing calcium hydride:

$$CaH_2 + 2\ H_2O \rightarrow Ca(OH)_2 + 2\ H_2\uparrow$$

2. Review of Properties of H_2 Relevant to its Use as Fuel

We review below some physical and chemical properties of hydrogen as they compare to two other fuels, methane and propane.

The most interesting property is of course the calorific value /10
of hydrogen, in the neighborhood of 28 Kcal/mole as opposed to 9 to 11 Kcal/mole for fossil fuels.

If the fact that a mole of hydrogen only weighs 2 g is taken into consdieration, the calorific value obtained per unit mass is very high. This partly compensates for hydrogen's very low density.

Table 1

		H_2	CH_4	C_3H_8
Standard Boiling Point	[°K]	20.3	111,7	230.8
Flamability Limits in Air, by Volume	[%]	4,1 - 14,8	5,3 - 15,0	2,3 - 9,5
Explosive Limits in Air by Volume	[%]	18,0 - 59,0	6,3 - 13,5	
Ignition Temperature	[°K]	850	807	736
Ignition Energy	[mJ]	0,02	0,3	0.25
Flame Temperature in Air	[°K]	2400	2190	2200
Flame Speed	[cm/s]	275	37	41

Table 2
Energy Values for Different Fuels [1]

		Heat of combustion	
		[KJ/g]	[KJ/cm³]
Liquid :			
hydrogen	(H_2)	124,7	8,7
methanol	(CH_3OH)	20,1	15,9
gasoline	(C_8H_{18})	44.3	30,9
Solid :			
vanadium hydride	(VH_2)	1,7	28,4
coal		32,2	41,8
wood		17,5	14,2
Gas :			
hydrogen	(H_2)	124,7	0,0010
methane	(CH_4)	61,1	0,0044

General Properties of Metal Hydrides

Metal hydrides, i.e. metal-hydrogen compounds, are generally grouped into three families according to the type of metal-hydrogen bond. Within each family, the physical and chemical properties are rather uniform [2,3,4,5].

The compounds are divided between the saline hydrides (primarily ionic bonding, NaH), the intermediate covalent hydrides (AlH_3), and the hydrides of transition metals (metallic type bonding, UH_3).

Hydride synthesis is exothermic. By virtue of Le Chatelier's laws concerning chemical equilibrium, production of hydrogen can be obtained by heating the hydride.

$$M + \frac{n}{2} H_2 \underset{2}{\overset{1}{\rightleftarrows}} MH_n + Q < 0$$

Increasing the temperature displaces the system in the direction of arrow 2, and therefore towards metal-hydrogen dissociation.

If an easily decomposable hydride with a high hydrogen content per unit mass could be synthesized, a "hydrogen sponge" would be obtained. It would be very flexible in use and its performance per unit mass would be comparable if not superior to that obtained with conventional methods of storage.

Table 3 gives an estimate of the level of hydrogen in some hydrides. /11

However, the chemistry of hydrogen-metal systems is not so simple. We have only spoken up to this point of binary compounds.

One must also survey the ternary compounds, $M_1M_2H_x$, the quaternary ones, $M_1M_2M_3H_x$, etc.

Table 3
Percentage of Hydrogen in Some Hydrides
(adapted from [7])

Hydride	Percent Hydrogen by weight	Wt. Hydride / Wt. Hydrogen
VH_2	3,7	26
MgH_2	7,6	13
~~K H~~	2,5	40
UH_3	1,2	80
ZrH_2	2,1	47
CaH_2	4,7	21
LiH	12,6	8
CeH_2	1,4	71

Kinetic and thermodynamic performance varies very rapidly as a function of the nature of the storage medium.

4. Basic Storage Media under Study

Four main approaches are presently being explored:
- Standard medium, $LaNi_5$ (Eindhoven, Battelle, DAM, ...)
- Mg medium (BNL, CEA, IFP, Battelle, ...)
- FeTi medium (BNL, CEA, Battelle, ...)
- Other media: VH_2, NbH_2, ...

4.1. $LaNi_5$ Medium

This clearly defined, ReM_5 type compound (Re = rare earth, M = transition metal), was the object of a very large number of experiments, particularly at Philips in Eindhoven.

Its interest resides mainly in its performance per unit volume since this compound is very dense and is represented by the formula $LaNi_5H_6$.

Table 4 gives some figures for $LaNi_5$ and other compounds

rich in hydrogen.

Table 4 /12
Comparison of Hydrogen Level for Some Compounds [8]

Compound	M [g/mole]	ρ [10^3 g/m^3]	N_H [10^{-28}/m^3]	%	ρ [10^3 g/m^3]
H_2O	18	1000	6,7	11	111
H_2SO_4	98,1	1841	2,2	2	36
Liquid CH_4	16,0	425	6,3	25	105
Liquid H_2	2	71	4,2	100	71
Ti H_2	49,9	3800	9,2	4	153
Zr H_2	93,2	5610	7,3	2,1	122
X H_2	90,9	3958	5,7	2,2	95
La H_2	140,9	5120	4,4	1,4	73
La H_3	141,9	5350	6,5	2,1	108
Ti Fe $H_{1,95}$	105,7	5470	6,2	1,85	101
La Ni_5H_6	438,5	6225	5,3	1,4	88

[Commas in tabulated material are equivalent to decimal points.]

Aside from the performance per unit mass of a hydride, the physical conditions of dissociation remain a basic element in the choice of medium.

While exploration of all ternary compounds is practically impossible, a study of the thermodynamic parameters is possible, particularly of enthalpy of reaction.

The general fromula can be written:

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$$\Delta H(AB_nH_{2m}) = \Delta H(AH_m) + \Delta H(B_nH_m) - \Delta H(AB_n)$$

In the case of $LaNi_5H_6$, one therefore writes:

$$\Delta H(LaNi_5H_6) = \Delta H(LaH_3) + \Delta H(Ni_5H_3) - \Delta H(LaNi_5)$$

This compound has a very great advantage from the thermodynamic point of view.

Its dissociation pressure (related to temperature by the equation $\log P = -\frac{\Delta S}{R} + \frac{\Delta H}{RT}$) is close to 3 bars at 40°C (figure 1).

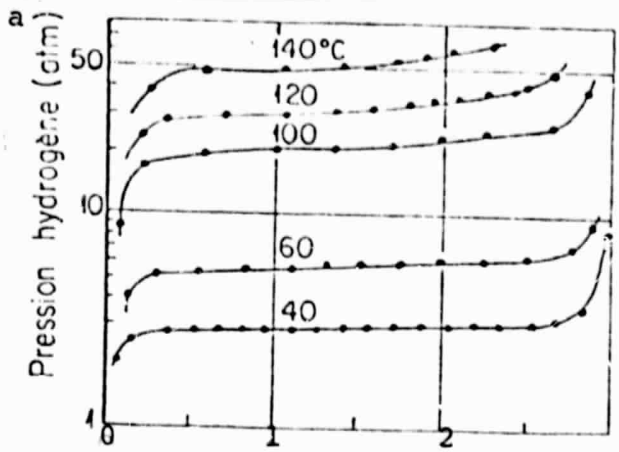

Figure 1

Key: a) Hydrogen Pressure (atm)

The hydride is thus very easily usable since it is sufficient to withdraw the hydrogen in the reservoir at the <u>ambient temperature</u> to make the system immediately produce more and maintain the equilibrium pressure.

Here again, however, this is much too simple. It is, as a matter of fact, possible to displace the isotherms in figure 1 practically at will by using compounds of the form $LaNi_4M$, where M is a transition metal other than Ni (figure 2). /13

The attractiveness of these compounds is thus readily apparent. The pressure can vary from 0.8 to approximately 8 bars at 40°C, as a function of the nature of M.

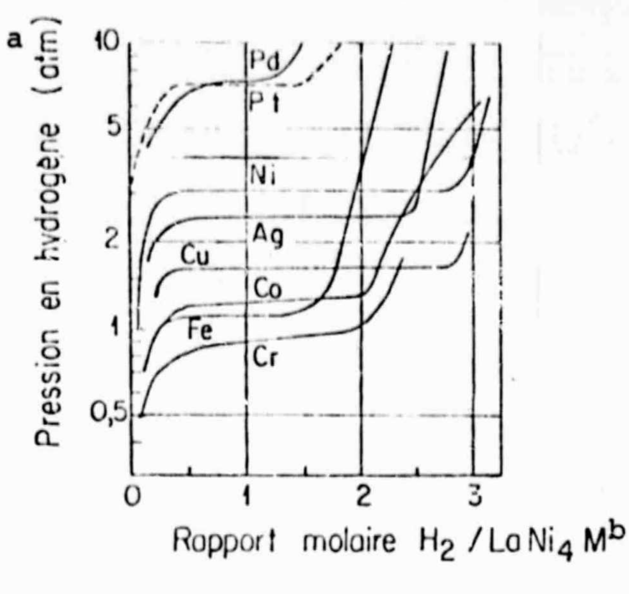

Figure 2
Pressure-Composition Isotherm for the Desorption of Hydrogen at 40°C for the Compound $LaNi_5$ and its Derivatives with the Formula $LaNi_4M$ (M = Pd, Ag, Cu, Co, Fe, Cr)

Key: a) Hydrogen Pressure (atm) b) Molar Ratio $H_2/LaNi_4M$

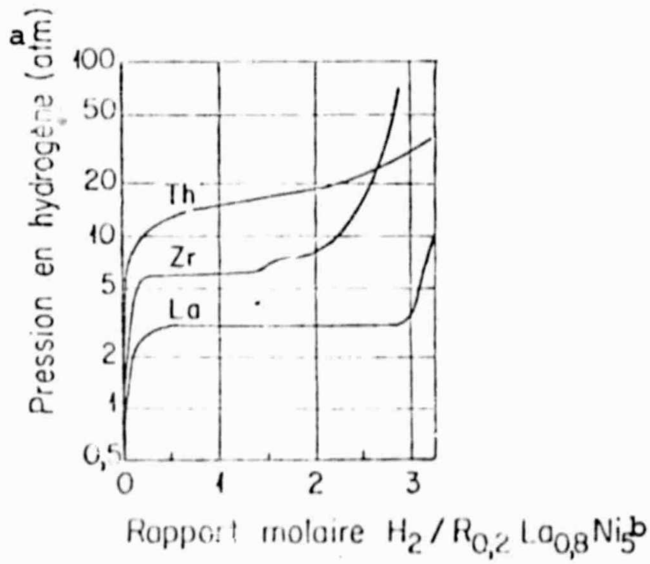

Figure 3
Desorption Isotherm at 40°C for $LaNi_5$ and its Derivatives $Zr_{0.2}La_{0.8}Ni_5$ and $Th_{0.2}La_{0.8}Ni_5$

Key: a) Hydrogen Pressure (atm) b) Molar Ratio $H_2/R_{0.2}La_{0.8}Ni_5$

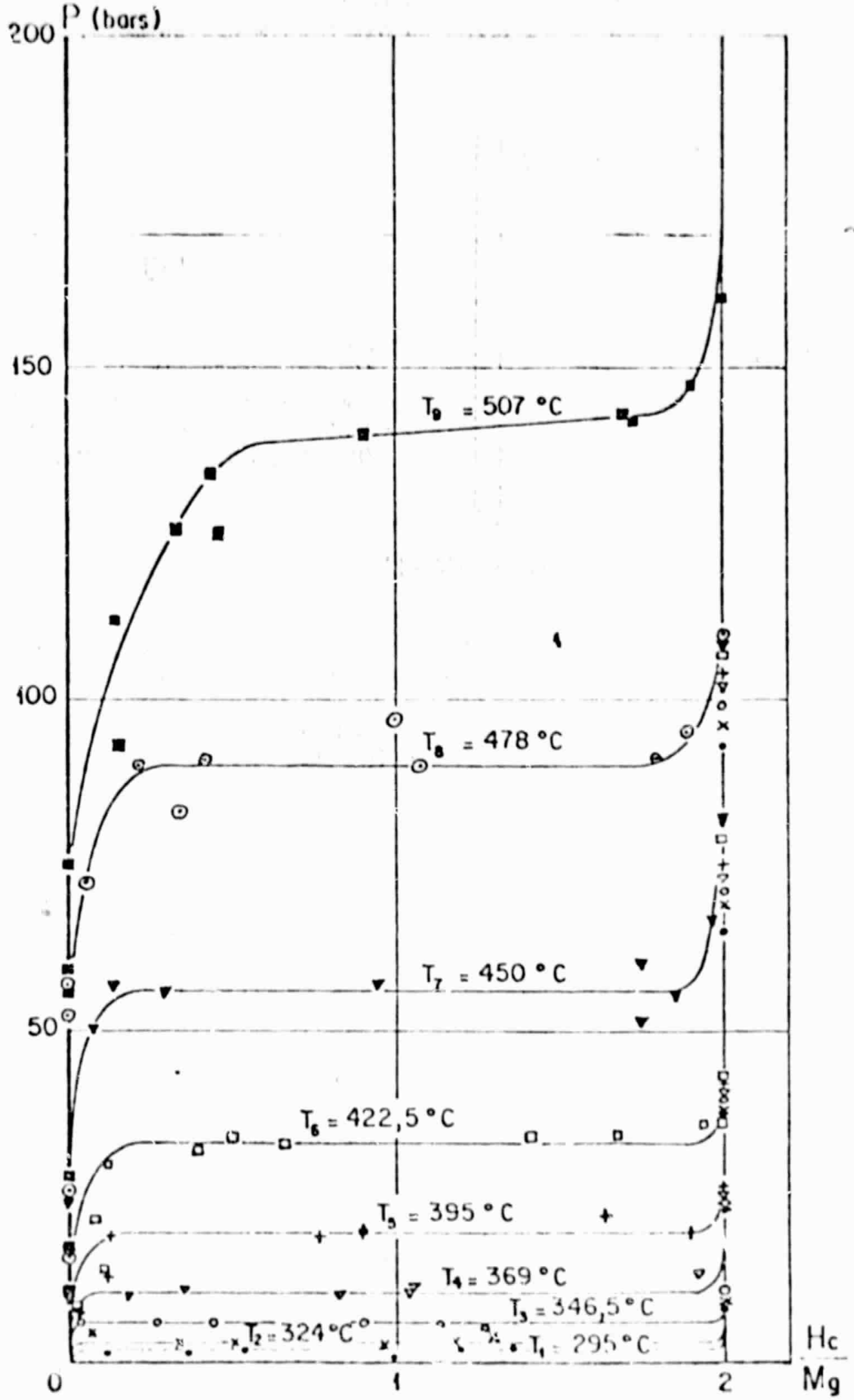

Figure 4
Equilibrium Pressure Isotherms -- Mg-H_2 System

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Similarly (figure 3), the five atoms of Ni can be conserved, and compounds of the form $R_{0.2}La_{0.8}Ni_5$ (R = Er, Y, Gd, Nd, Th, Zr) could be used.

4.2. Magnesium Medium

Magnesium hydride, MgH_2, behaves like a "true" chemical hydride.

The nature of the bond is fairly well determined and the isotherms are rectangular (figure 4).

MgH_2 is very attractive from the point of view of mass since /15
its hydrogen content is about 7.7% by weight. The application we have chosen, a fuel tank on board an automobile, makes this figure very improtant. Our work has therefore concentrated on magnesium and its derivatives [9, 10].

Many experiments, carried out with several grades of magnesium and with hydrogen of varying purity (tables 5 and 6) have allowed us to determine the conditions of synthesis for MgH_2 (figures 5, 6, and 7).

Our study documented the problem of aging of the metal storage medium as a result of oxidation (figure 7).

To get around this difficulty and obtain at the same time a lower enthalpy of reaction (-17.8 Kcal/mole H_2 for MgH_2) the compounds defined as Mg_2Cu and Mg_2Ni were hydrided.

While the perfomance per unit mass diminished, the behavior over time turned out to be much improved. We carried out more than twenty cycles on Mg_2Cu without observing any degradation (figure 8).

The enthalpy of reaction proved to be more accessible. It was -15.5 Kcal/mole H_2 for Mg_2Cu and -13.8 Kcal/mole H_2 for Mg_2Ni (figure 9).

Table 5
Sepcifications of Magnesium Powders in Use
(Manufacturer's Data: SA Baudier & Fils, Sénécourt, 60 Liancourt)

Powder Reference No.		20/50	50/100	DA 200	80/200
Apparent Density	[g/cm^3]	0,8	0,8	0,7	0,6
Compacted Density	[g/cm^3]	1	1	0,8	0,7
Granulometry	[micron]	400-1000	150-400	70-200	10-80
Av. Diameter	[micron]	-	-	15	10
Specific Surface	[cm^2/g]	-	-	2300	3500
Moisture	[%]	< 0,05	< 0,05	< 0,05	< 0,05
Grease	[%]	< 0,02	< 0,02	< 0,02	< 0,02
Nonmetallic Impurities	[%]	< 0,1	< 0,1	< 0,1	< 0,1
Mg	[%]	≥ 97	≥ 97	≥ 99	≥ 99
Al	[%]	≤ 1,5	≤ 1,5	-	-
O_2	[%]	< 0,15	< 0,15	< 0,15	< 0,15
Cl_2, Mn, Si	[%]	< 0,2	< 0,2	< 0,2	< 0.2

Table 6
Purity of U and N55 Hydrogen

	$H_2 + D_2$ [%]	O_2 [ppm]	H_2O [ppm]	N_2 [ppm]	CH_4 [ppm]	D_2 [ppm]
U	> 99,995	< 5	< 5	< 40	-	-
N 55	> 99,9995	< 1,5	< 2	< 2,5	< 0.1	≈ 150

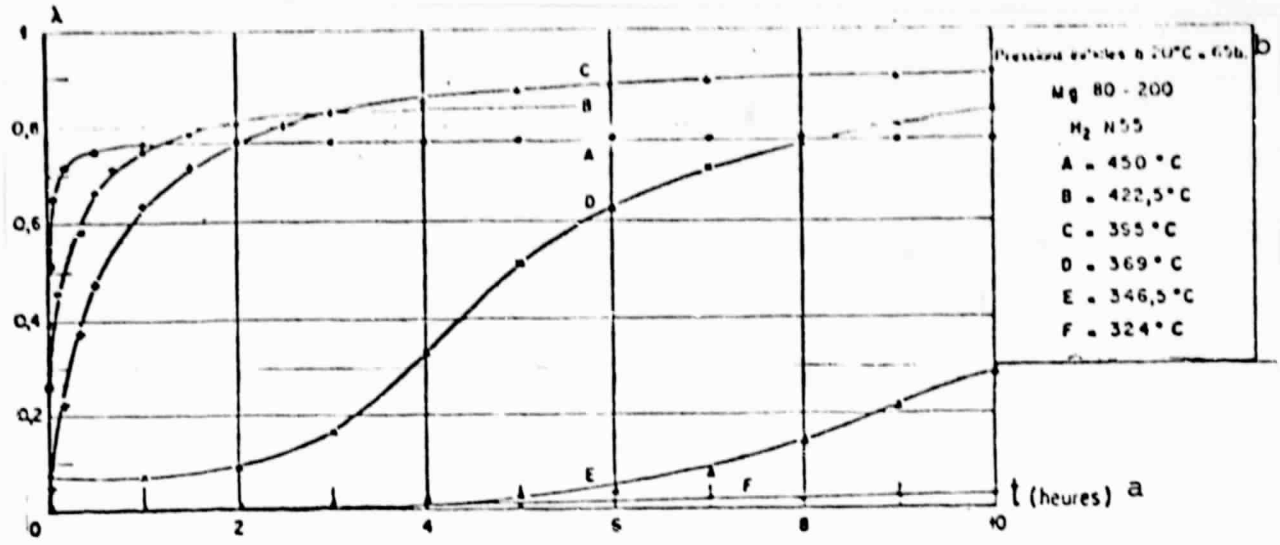

Figure 5
Influence of Temperature on the Synthesis of MgH_2
Degree of Advancement λ = f(time)

Key: a) (hours)
b) Initial Pressure at 20°C = 65 bar

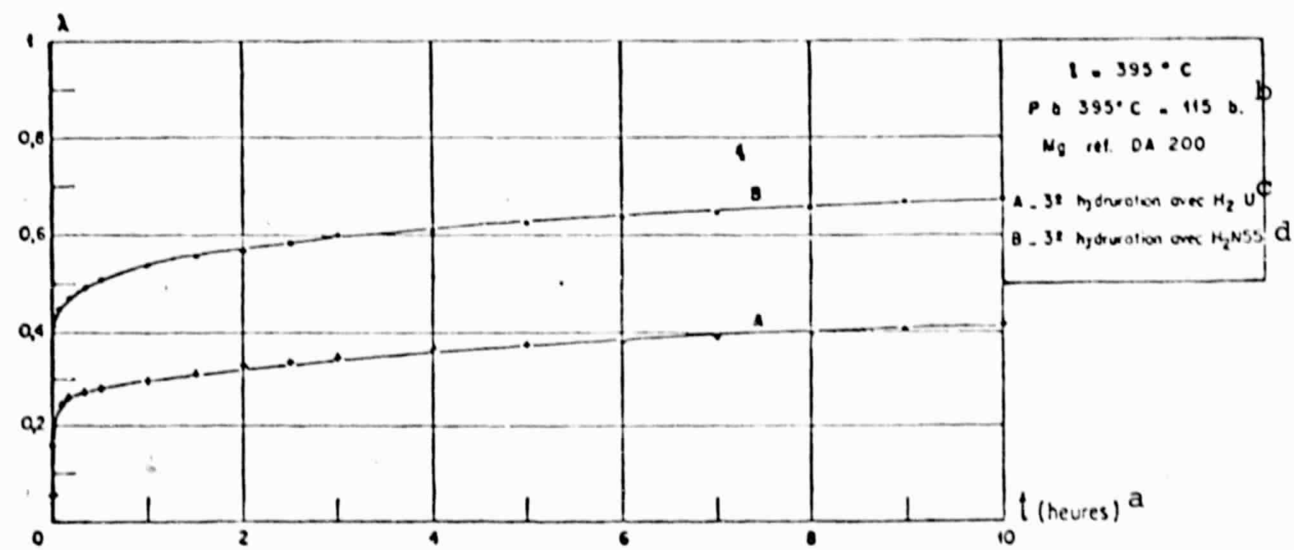

Figure 6
Influence of Hydrogen Purity on the Synthesis of MgH_2
Degree of Advancement λ = f(time)

Key: a) (hours)
b) P at 395°C = 115 bar
c) hydridation with U H_2
d) hydridation with N55 H_2

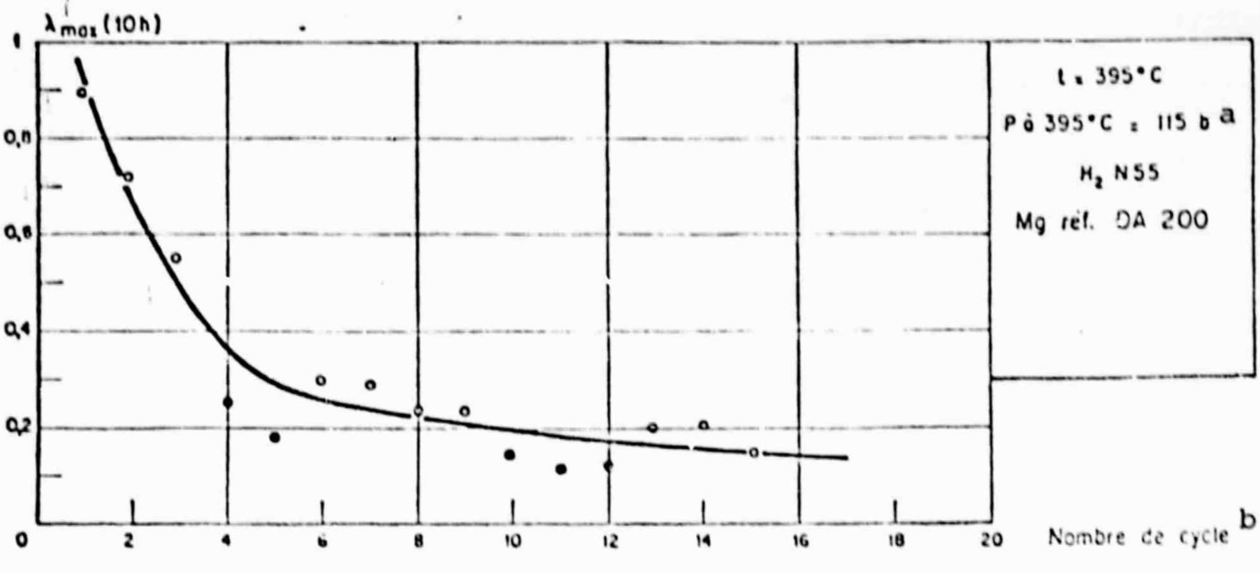

Figure 7
[Caption Missing in Original Copy]

Key: a) P at 395°C = 115 bar
b) Number of Cycles

/17

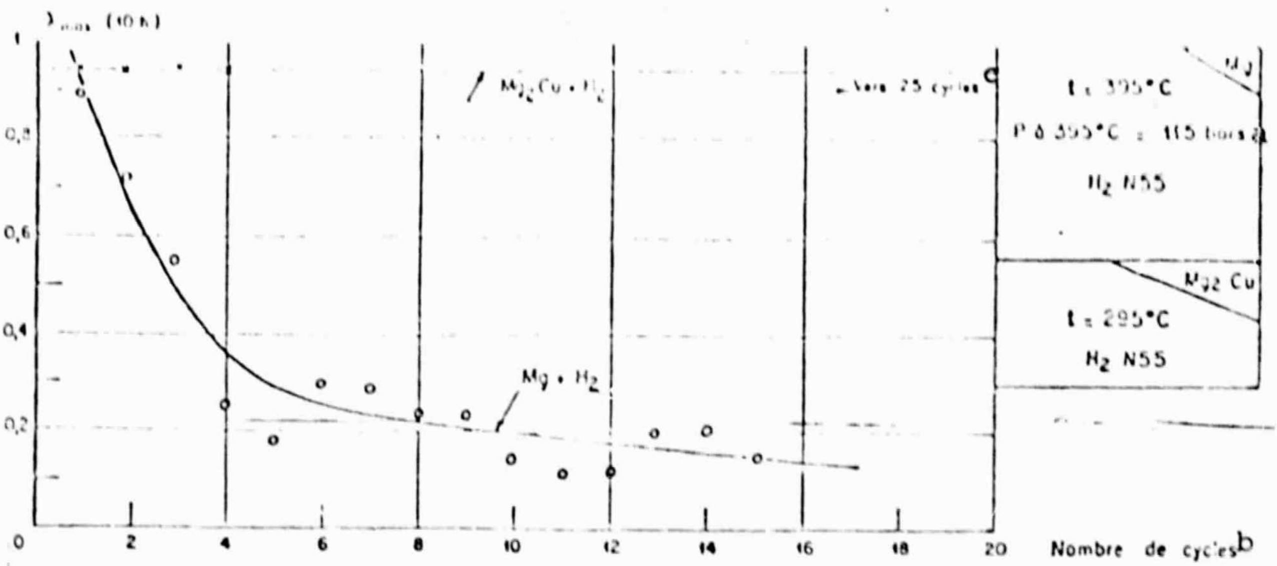

Figure 8
Synthesis of Magnesium Hydride
Degree of Advancement in 10 h = f(number of cycles)

Key: a) P at 395°C = 115 bar
b) Number of Cycles
c) towards 25 cycles

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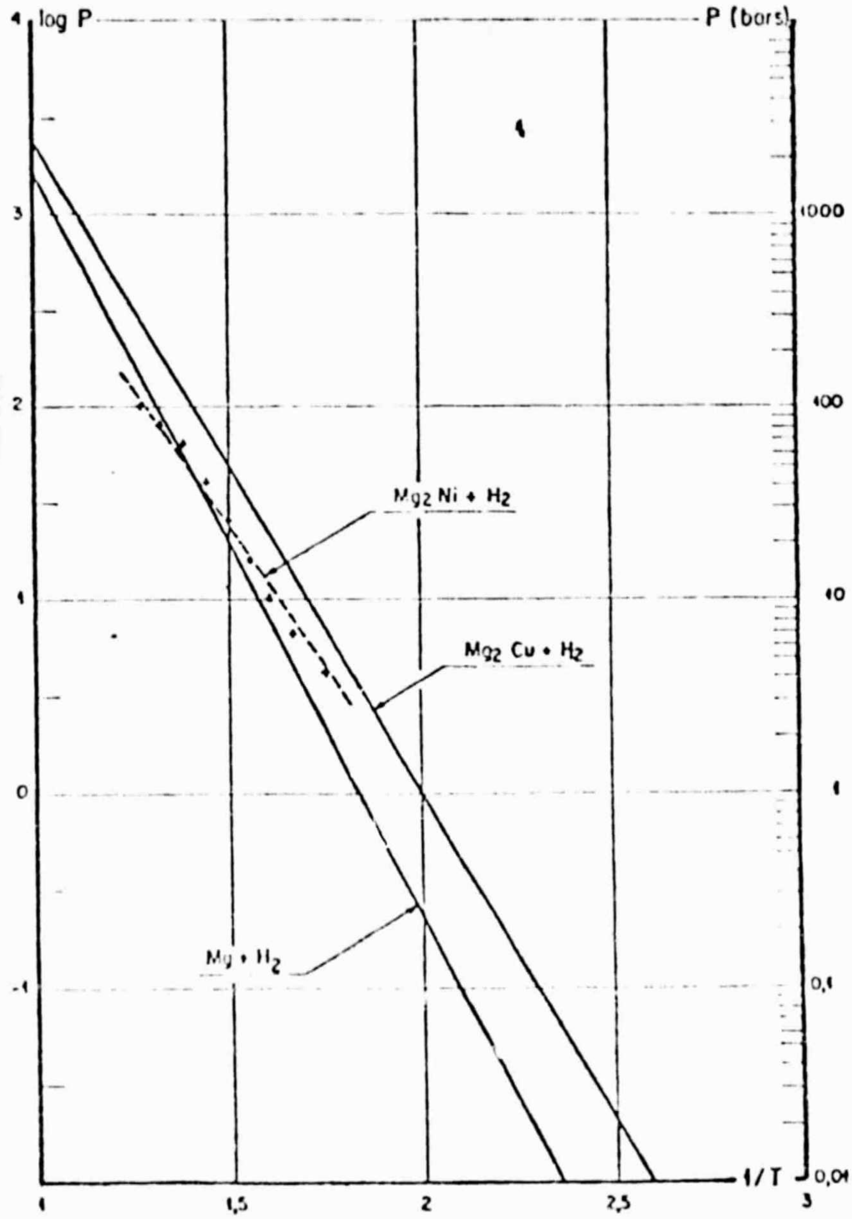

Figure 9
Log P = f(1/T)

4.3. FeTi Medium

The stoichiometric formula $FeTiH_2$ exhibits an attractive performance per unit mass, with about 2% hydrogen by weight.

However, we are confronted with a transition metal. On the one hand, nonstoichiometric problems appear. On the other, the isotherms are not as rectangular as for magnesium, for example (figure 10).

Be that as it may, the thermodynamic characteristics of this system make it the leader for the design of storage containers [11, 12, 13, 14].

Figures 10 to 12 were taken form the publications cited above and reveal the sensitivity of these systems, just as was shown for $LaNi_5$.

First of all, the oxygen content can displace an isotherm, by 1 bar, for example (figure 11).

Then, it is possible to introduce transition elements such as Cr, Mn, and Co into the FeTi. This changes the form of the isotherms considerably, as depicted in figure 12.

Here again, the curve can be displaced by a decade, when shifting from FeTi to $TiFe_{0.9}Ni_{0.1}$.

5. Problems Posed by a Hydride Storage Container

We have experimented with two hydrogen-generating hydride reservoirs (Project G2H).

The first, containing 0.3 Nm^3 of hydrogen, used $SmCo_5$ (an isotype of $LaNi_5$).

Its design, which was very simple made it not very practical.

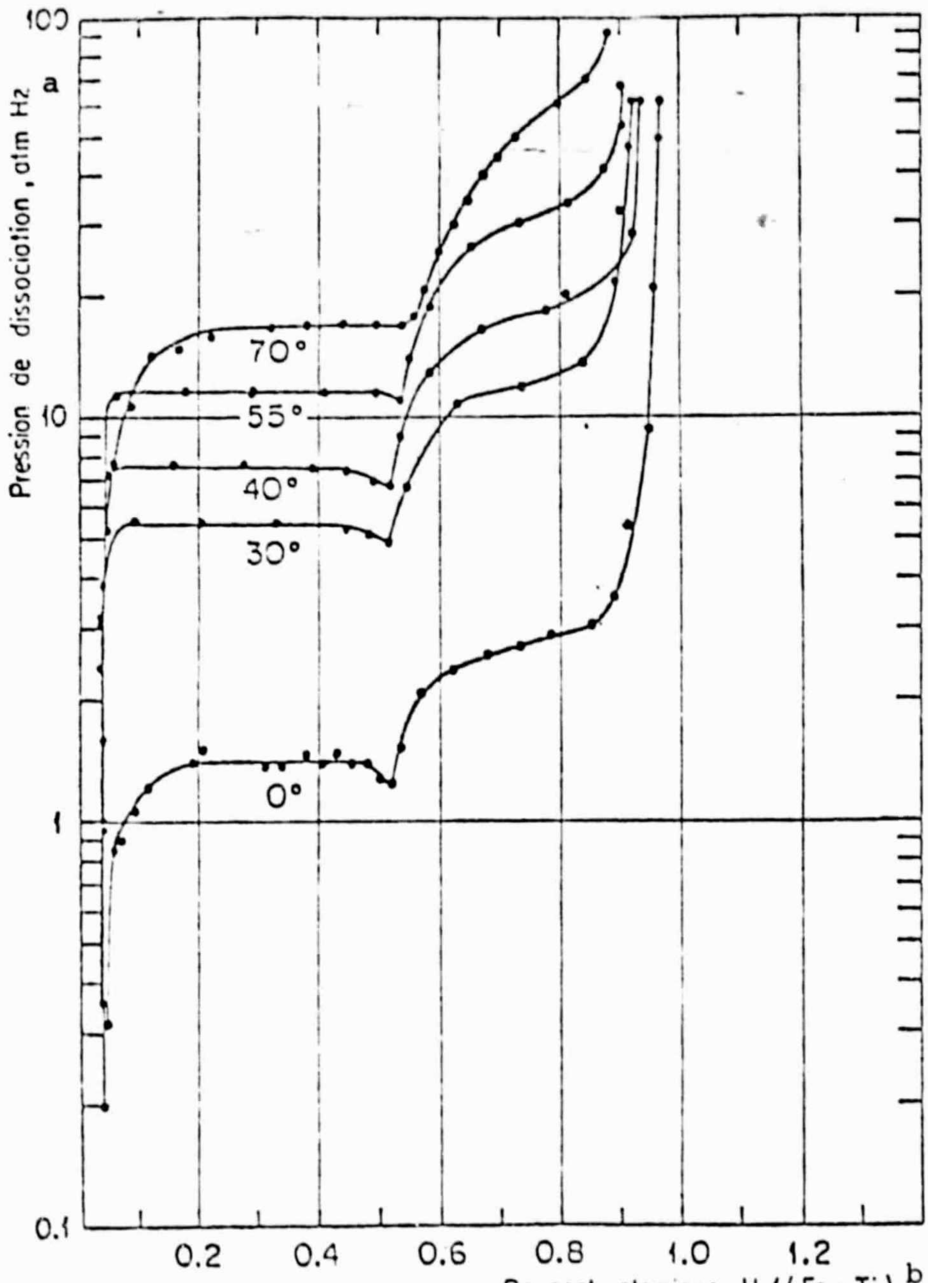

Figure 10
Pressure-Composition Isotherms for the FeTi-H_2 System

Key: a) Dissociation Pressure, atm H_2
b) Atomic Ratio H/(Fe + Ti)

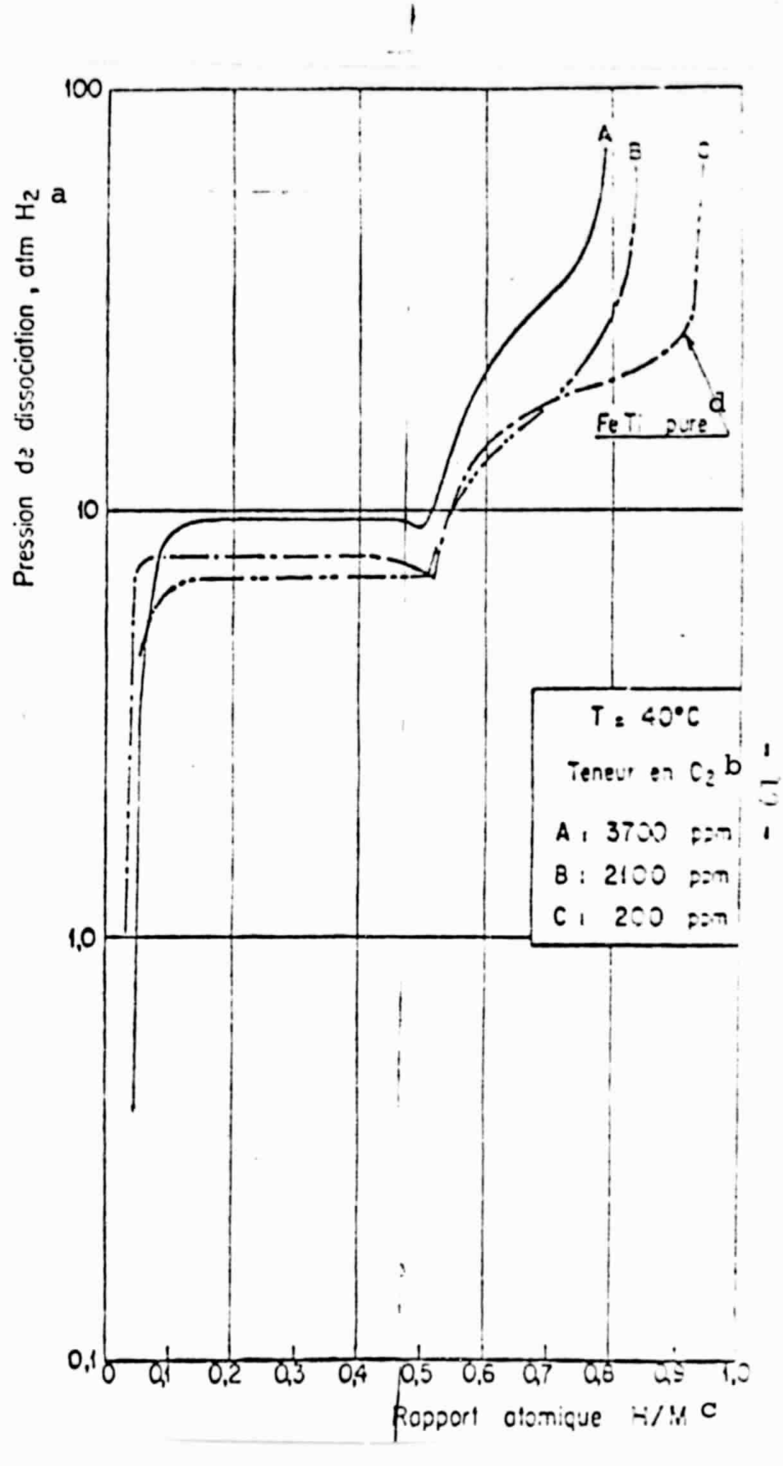

Figure 11
Effect of O_2 Content on the FeTi + H_2 System

Key: a) Dissociation Pressure, atm H_2
b) O_2 Content
c) Atomic Ratio H/M
d) Pure FeTi

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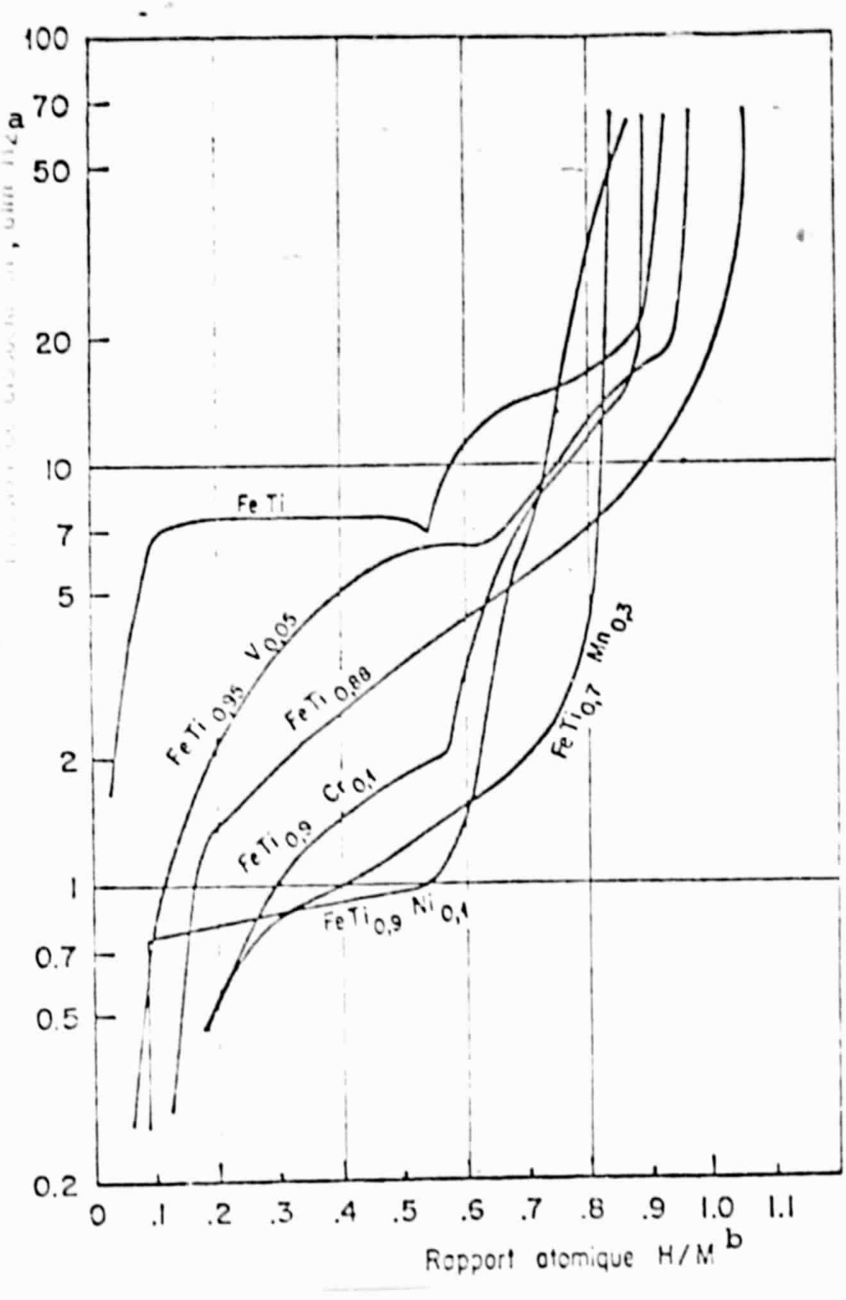


Figure 12
The Effect on the 40°C Desorption Isotherm
of Substituting Iron for Various Transition Metals in FeTi
[sic]

Key: a) Dissociation Pressure, atm H_2
b) Atomic Ratio H/M

The second reservoir, containing 3 Nm^3 of hydrogen, was filled with 20 kg of $LaNi_5$ (figure 13). It functioned satisfactorily. The problems wich arose were the following:

- dead space in the reservoir,
- effect of settling,
- diffusion of hydrogen in the powder,
- thermal exchanges,
- aging of medium in the course of the charge-discharge cycles.

As an example, a characteristic curve for charging is given in figure 14.

Studies are in progress for constructing a reservoir resolving these problems and capable of storing 10 kg of hydrogen using FeTi. In addition, a reservoir containing 2 kg of hydrogen by means of Mg and its alloys is under development.

6. Combustion of Hydrogen Generated by Hydrides in Thermal Engines

One obvious application is the use of hydrogen as a fuel for propulsion, particularly on the ground.

This problem has two aspects: First of all is the design of /22 the on-board tank. Then there is the combustion itself in conventional thermal machines.

An automotive hydride container has to meet certain criteria (weight, bulkiness, autonomy, safety, recharging, etc.) which make its development almost impossible before the final choice of hydride is made.

That is why we are interested in the second part: "combustion of hydrogen".

Our experiments were performed first on a single cylinder "Bernard" engine, then on a Renault R4 automobile, and finally on a

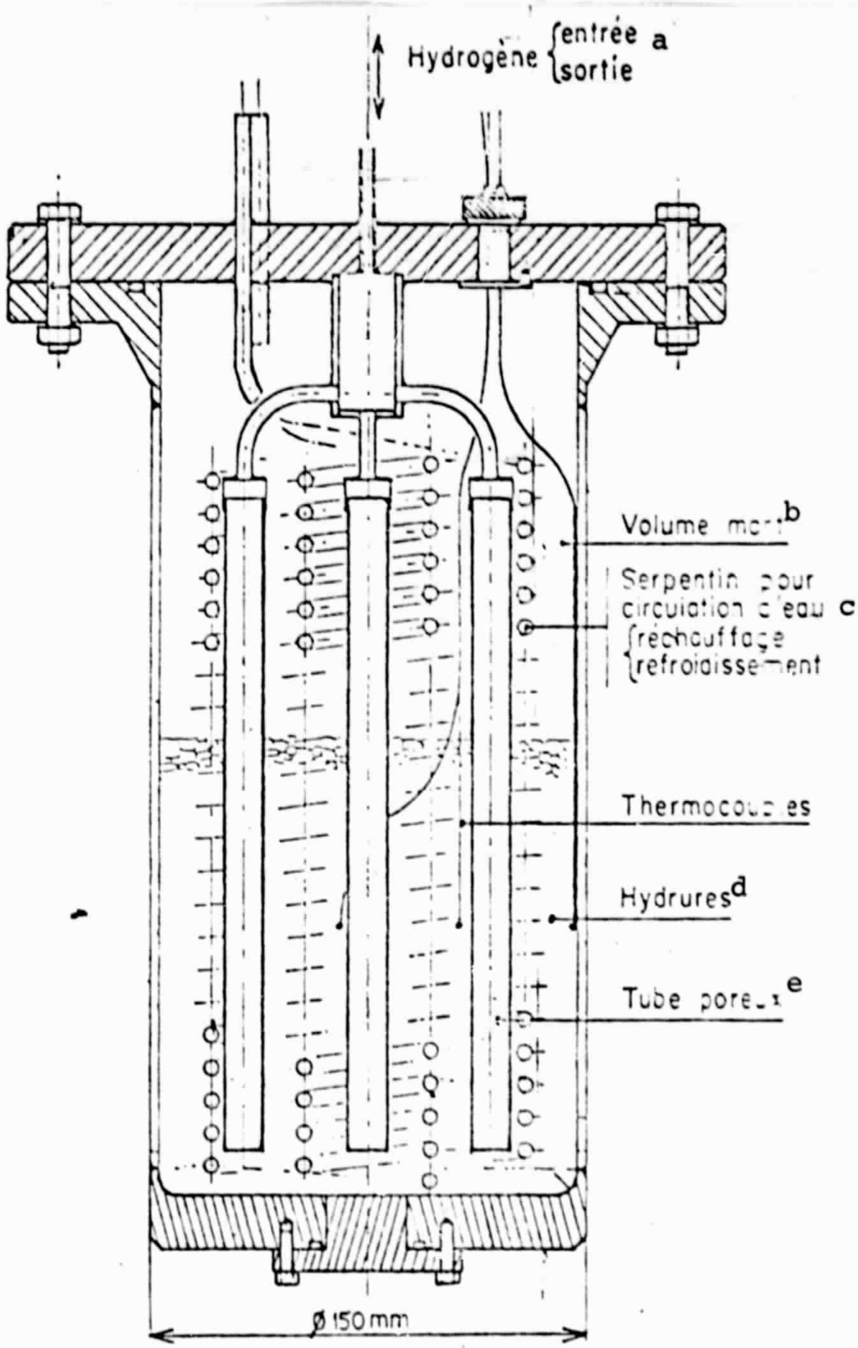


Figure 13
Schematic of a Hydride-Based Hydrogen Generator
(Project G_2H, 20 kg of $LaNi_5$)

Key: a) Hydrogen (Intake, Exhaust)
b) Dead Space
c) Coil for Circulation of Water (Heating, Cooling)
d) Hydrides
e) Porous Tube

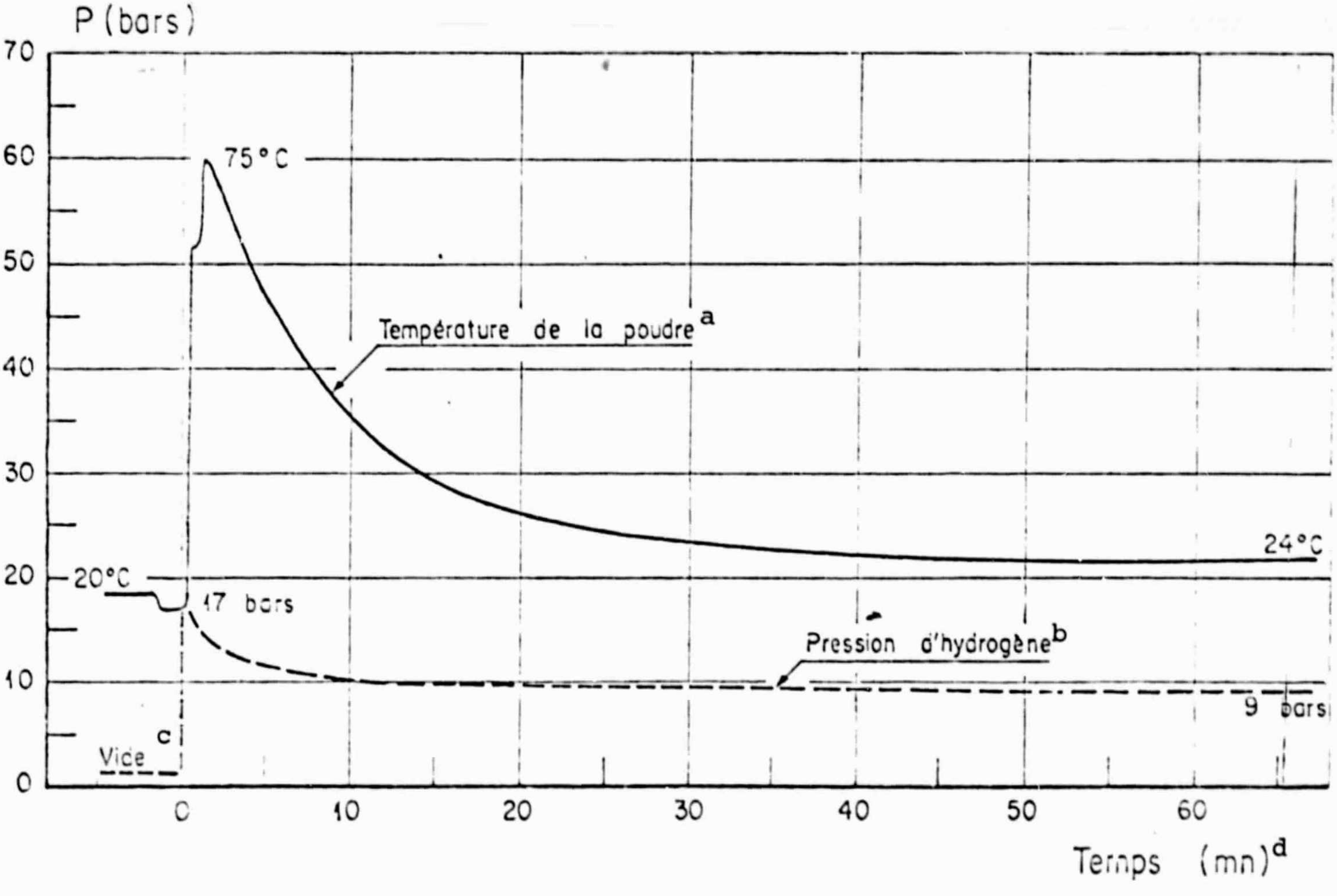


Figure 14
Hydrogen Generator, 20 kg $LaNi_5$, Loading Curve (Fifth Hydridation)

Key: a) Temperature of Powder c) Empty
b) Pressure of Hydrogen d) Time (min)

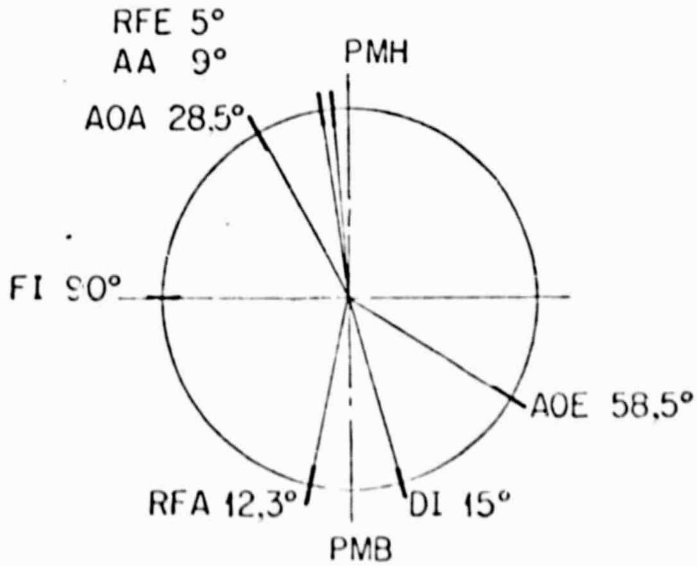
250 hp turbine built by the Société des Bennes Marrel.

6.1. "Bernard" Engine

This is a single cylinder engine originally designed for use with a gasoline-air mixture. Reference measurements were made with gasoline. The maximum power was 2.35 hp (1.73 kw).

Hydrogen was directly injected into the combustion chamber by means of an injector placed on the cylinder head (water-cooled). (See photograph 1.)

Ignition was performed by the storage battery - coil - distributor circuit. This network allowed very fine adjustments to be made (photograph 2). Different engine timings were tried out. Optimum performance was obtained with the diagram below:



PMH = Top Dead Center
AOE = Exhaust Opening Advance
DI = Injector On
PMB = Bottom Dead Center
RFA = Intake Closing Lag
FI = Injector Off
AOA = Intake Opening Advance
AA = Ignition Advance
RFE = Exhaust Closing Lag

The range of engine speeds was from idle to 3600 rpm. The power collected by the dynamo-brake (photograph 3) was dissipated in a field of incadescent lights.

Figures 15 to 21 give the principal results obtained.

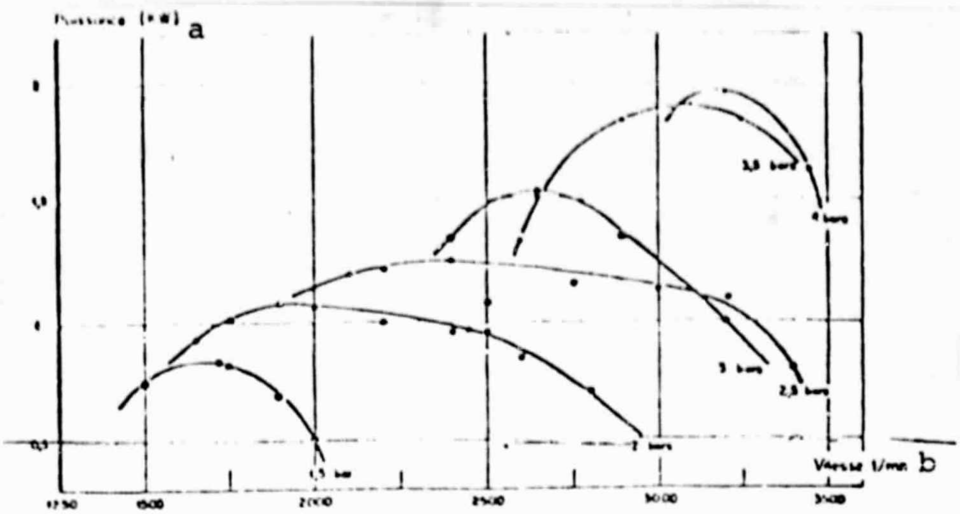


Figure 15
Corrected Power Curves
Power = f(speed, injection pressure)

Key: a) power (kw) b) speed (rpm)

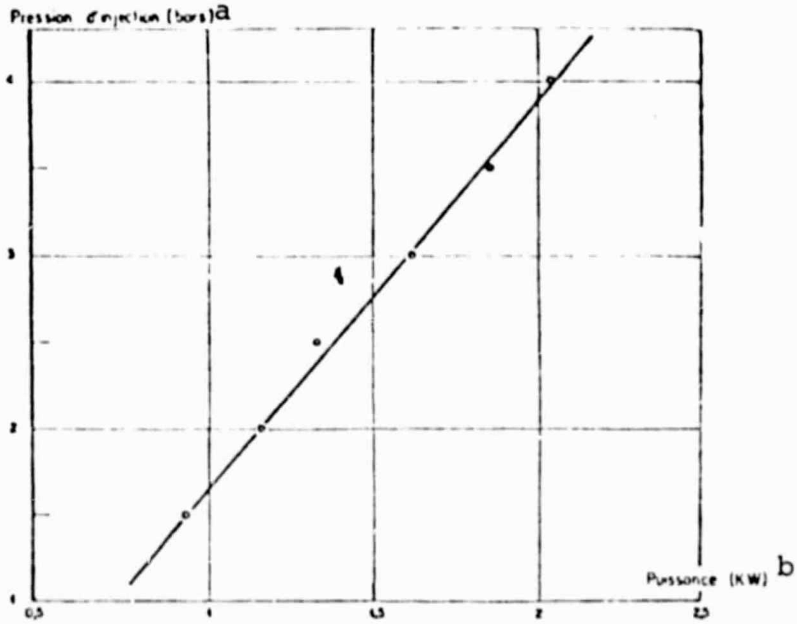


Figure 16
Injection Pressure = f(power)

Key: a) Injection Pressure (bars) b) Power (kw)

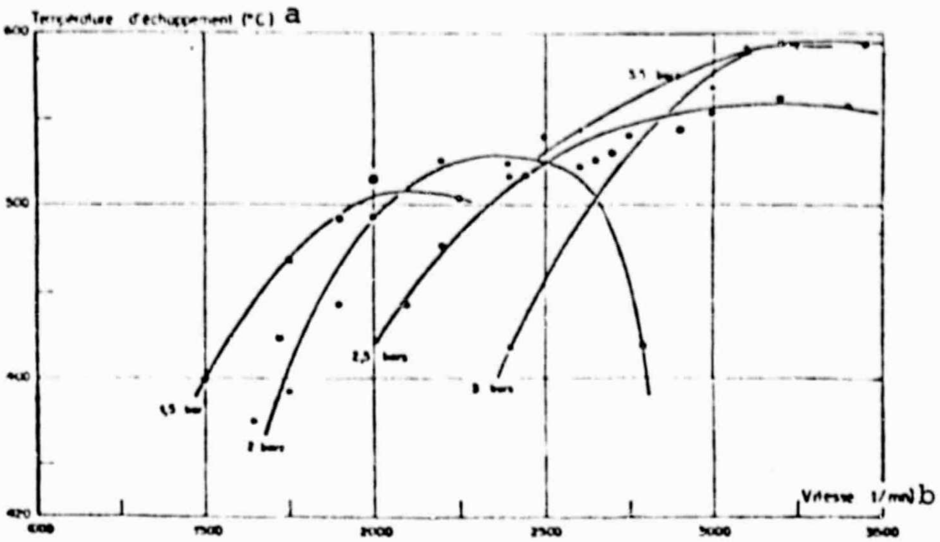


Figure 17
Exhaust Temperature = f(speed, injection pressure)

Key: a) Exhaust Temperature (°C)   b) Speed (rpm)

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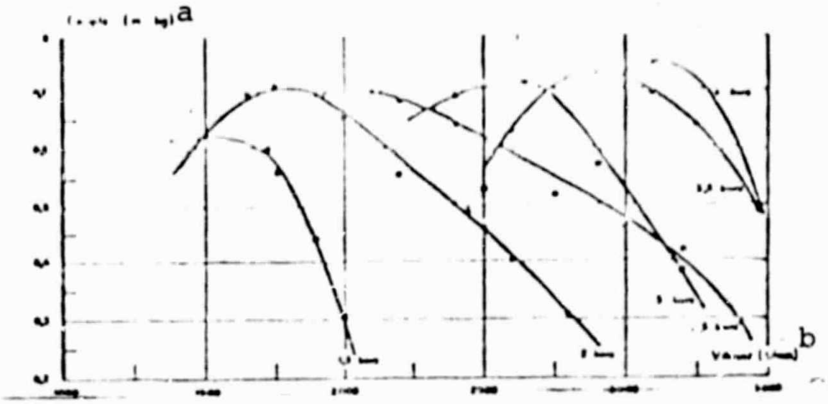


Figure 18
Torque = f(speed, injection pressure)

Key: a) Torque (mkg)   b) Speed (rpm)

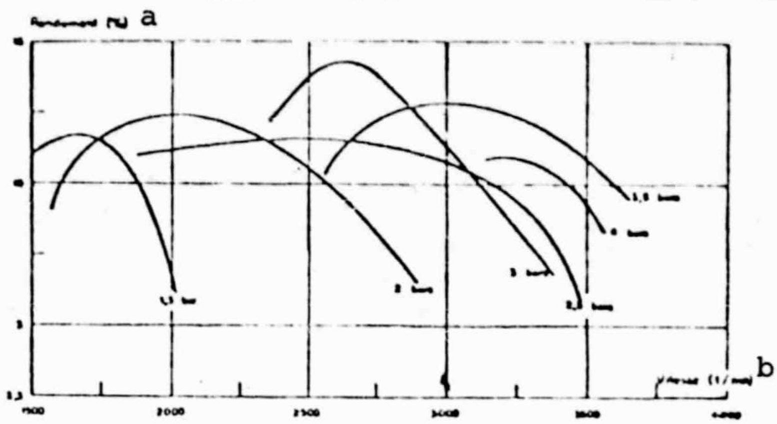


Figure 19
Efficiency = f(speed, injection pressure)

Key: a) Efficiency (%) b) Speed (rpm)

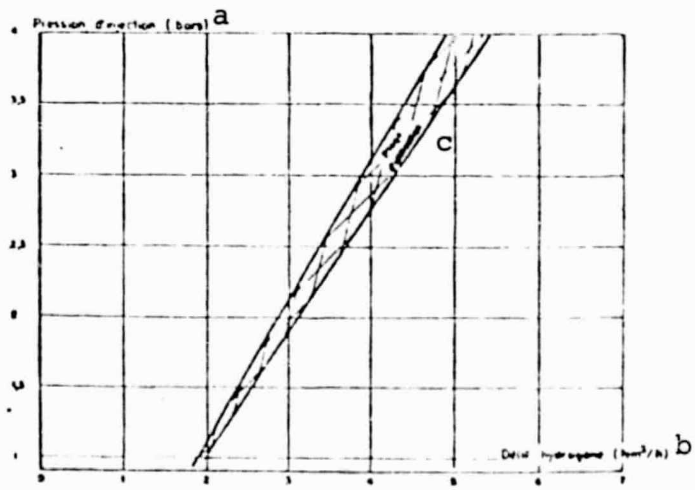
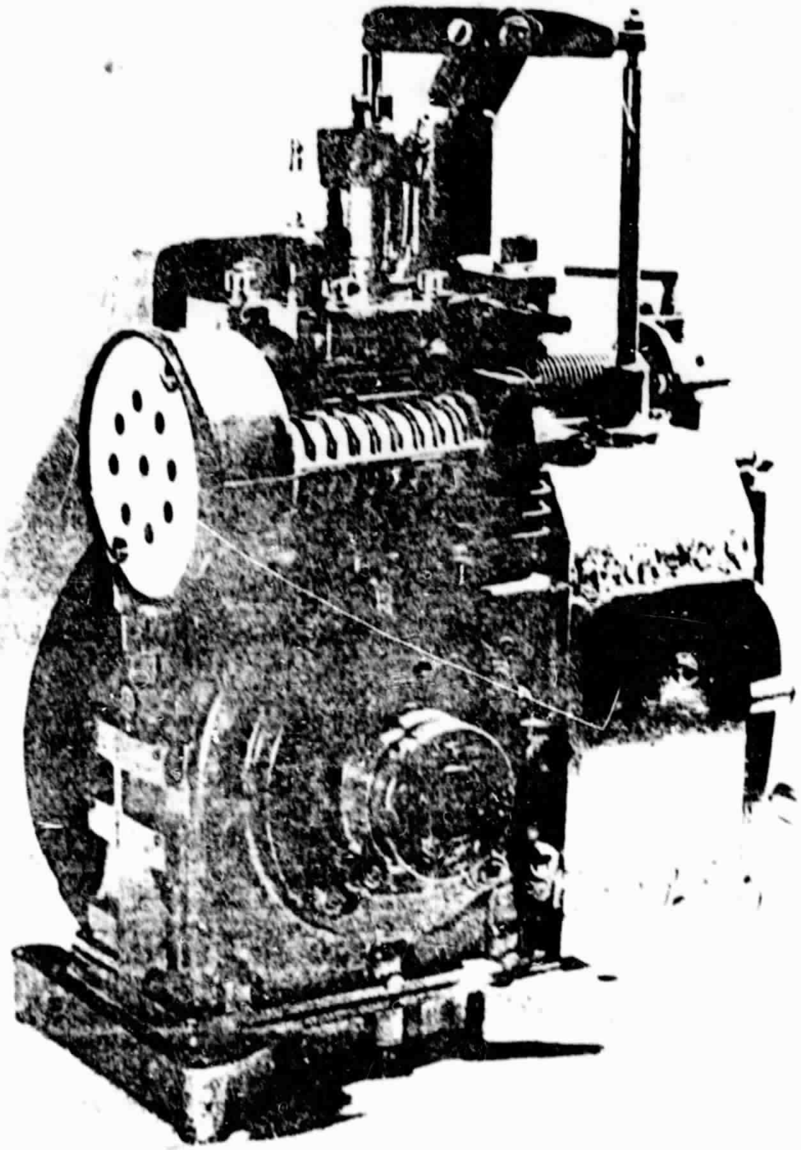
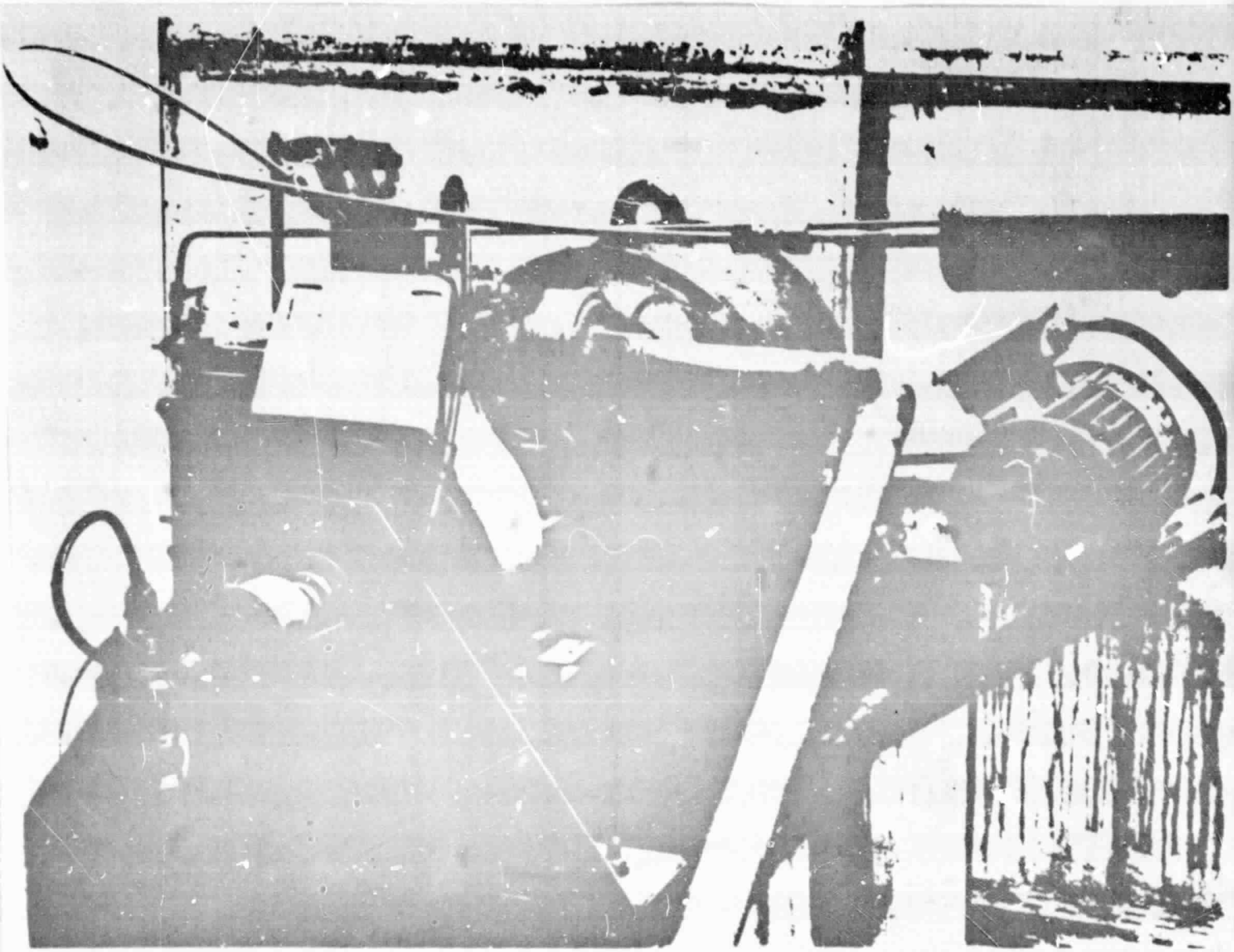


Figure 20
Injector Characteristics
Injection Pressure = f(hydrogen flow)

Key: a) Injection Pressure (bars) c) Range of [illegible]
b) Hydrogen Flow (Nm^3/h)



Photograph 1
"Bernard" Engine Adapted for Direct Hydrogen Injection



Photograph 2
Partial View of Setup

In the foreground is the ignition coil and distributor, then the engine with the injector on top, and finally in the rear is the dynamo-brake and, at right, the electric starter.

Photograph 3
Measurement Stand

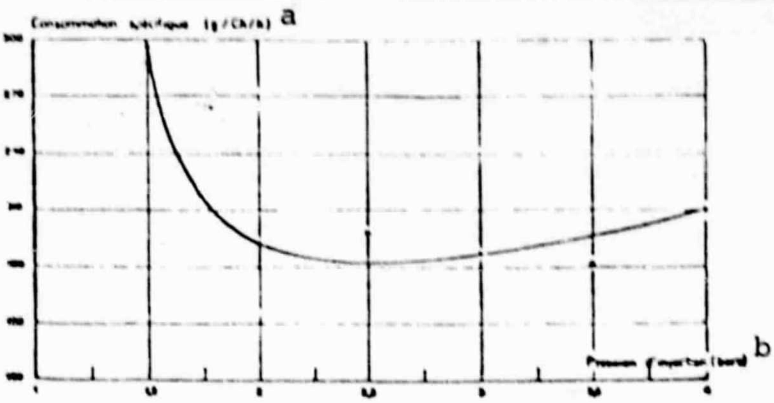


Figure 21
Specific Fuel Consuption = f(injection pressure)

Key: a) Specific Fuel Consumption (g/hp/h)
b) Injection Pressure (bars)

Note that the specific fuel comsumption is close to 200 g/hp/h /27
at 3.5 bars. When hydrogen's calorific value per unit mass is taken into account, this figure might appear high. In fact, when the engine ran on gasoline, a specific fuel consumption of 540 g/hp/h was observed. These two values are well within the ratio of calorific values (2.7). The overall efficiency of the engine was therefore not degraded. It remained very low (figure 19) since it attained only 15%.

6.2. Renault R4 Automobile

We have built a hydrogen-air supply system for an automobile with a four cylinder, 35 hp engine.

The only two modifications concerned the carburator and the engine timing.

The hydrogen-air mixture was supplied by an "Impco" gas carburetor. The exhaust gas was recirculated so as to impoverish the mixture (photograph 4). In addition, nitrogen plays an inhibitory role in hydrogen-air mixtures.

Photograph 4
Hydrogen Powered "Renault 4L"

The engine timing was changed simply by shifting the rocker arm clearances. There was no thought of adapting a fuel injection system to an already very complex cylinder head.

Also, the performance remained quite low, 60 km/h.

6.3. Hydrogen Turbine

In collaboration with the Société des Bennes Marrel, we undertook the construction of a fuel supply for a gas turbine built by the company. We first of all supplied only one of the turbine's two burners (figure 22 and photograph 5).

The recorded results were very satisfactory. Turbine operation required a maximum flow of 5.6 g H_2/sec for a turbine of 250 hp without heat exchanger.

Since this experiment was undertaken only as a preliminary test, no measurement of power was made.

7. Conclusion

The storage of hydrogen in the form of metallic hydrides is presently confronted with two problems.

One completely metallurgical problem is the preparation of very high purity binary or ternary compounds (necessary for having complete control of the reactions). This problem becomes even more delicate when large production runs are concerned.

A technical problem is the design of the storage containers, which can rapidly become heavy and complicated: filters, heat exchangers, accelerator pump, heating systems, stirring, etc. It is unlikely that recharging can occur outside the factory (exchange of tank) because of the energy involved and the time required to exchange that energy.

Finally, a problem concerning energy policy can be cited since hydrogen is intimately tied to nuclear power.

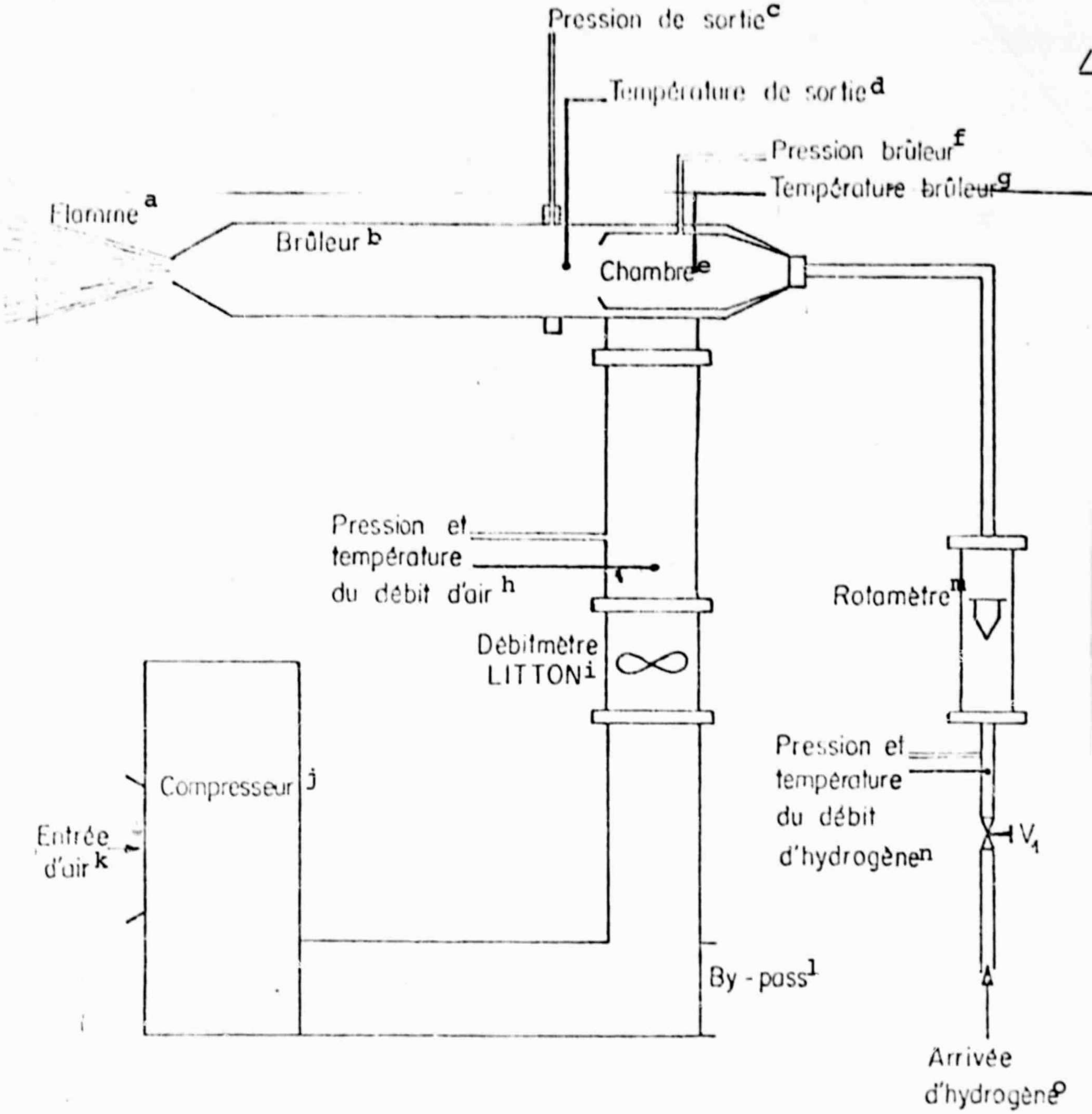


Figure 22
General Schematic Drawing of Experimental Setup

Key:
- a) Flame
- b) Burner
- c) Exit Pressure
- d) Exit Temperature
- e) Chamber
- f) Burner Pressure
- g) Burner Temperature
- h) Pressure and Temperature of Air Flow
- i) Litton Flowmeter
- j) Compressor
- k) Air Intake
- l) Bypass
- m) Rotameter
- n) Pressure and Temperature of Hydrogen Flow
- o) Hydrogen Intake

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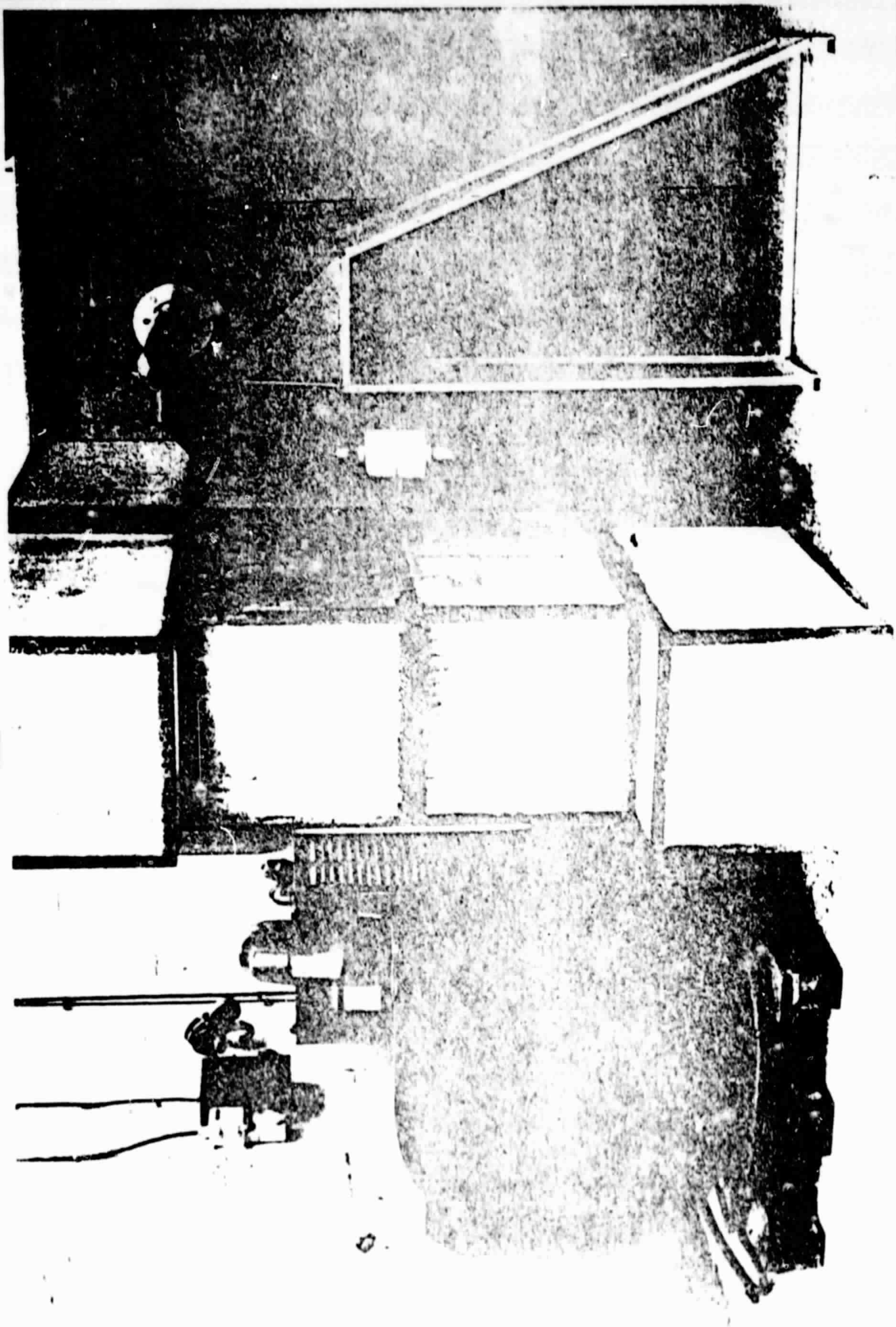

Photograph 5
Test of the Burner in the "Bennes-Marrel" 250 hp Hydrogen Turbine

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